



# STIC Search Report

## EIC 1700

STIC Database Tracking Number: 113341

**TO: Joseph Anthony**

**Location:** 10D21

**Art Unit : 1714**

**February 4, 2004**

**Case Serial Number: 09/786719**

**From: Michael Newell**

**Location: EIC 1700**

**REMSEN 4A30**

**Phone: 571/272-2538**

**MNewell@uspto.gov**

### Search Notes

Deliver to Remsen 10D21.

There are three sets of results. The first is art by a combination of the inventors, searched in HCAPLUS. The second set resulted from the structure search, using L5 as the query structure. The third set was derived from a search of phosphonamidic acids and ammonia salts/compounds, using a combination of registry numbers and text terms, then limited to art prior to 2000.

Access DB# 113341**SEARCH REQUEST FORM**

Scientific and Technical Information Center

Requester's Full Name: Joseph Anthony Examiner #: 68496 Date: 2/03/04  
Art Unit: 1714 Phone Number 30 \_\_\_\_\_ Serial Number: 091786719  
Mail Box and Bldg/Room Location: 10021 Results Format Preferred (circle): PAPER DISK E-MAIL

**If more than one search is submitted, please prioritize searches in order of need.**

\*\*\*\*\*

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: \_\_\_\_\_

Inventors (please provide full names): \_\_\_\_\_

Earliest Priority Filing Date: \_\_\_\_\_

*\*For Sequence Searches Only\* Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.*

\*\*\*\*\*

**STAFF USE ONLY**

	Type of Search	Vendors and cost where applicable
Searcher: <u>M. Newell</u>	NA Sequence (#) _____	STN <u>709.17</u>
Searcher Phone #: <u>571-272-2538</u>	AA Sequence (#) _____	Dialog _____
Searcher Location: <u>Rem 4A30</u>	Structure (#) <u>1</u>	Questel/Orbit _____
Date Searcher Picked Up: <u>2/4/04</u>	Bibliographic <u>✓</u>	Dr.Link _____
Date Completed: <u>2/4/04</u>	Litigation _____	Lexis/Nexis _____
Searcher Prep & Review Time: <u>60</u>	Fulltext _____	Sequence Systems _____
Clerical Prep Time: _____	Patent Family _____	WWW/Internet _____
Online Time: <u>120</u>	Other _____	Other (specify) _____



# STIC Search Results Feedback Form

**EIC17000**

Questions about the scope or the results of the search? Contact *the EIC searcher or contact:*

Kathleen Fuller, EIC 1700 Team Leader  
571/272-2505 REMSEN 4B28

## Voluntary Results Feedback Form

➤ I am an examiner in Workgroup:  Example: 1713

➤ Relevant prior art **found**, search results used as follows:

- ☐ 102 rejection
- ☐ 103 rejection
- ☐ Cited as being of interest.
- ☐ Helped examiner better understand the invention.
- ☐ Helped examiner better understand the state of the art in their technology.

*Types of relevant prior art found:*

- ☐ Foreign Patent(s)
- ☐ Non-Patent Literature  
(journal articles, conference proceedings, new product announcements etc.)

➤ Relevant prior art **not found**:

- ☐ Results verified the lack of relevant prior art (helped determine patentability).
- ☐ Results were not useful in determining patentability or understanding the invention.

**Comments:**

Drop off or send completed forms to EIC1700 REMSEN 4B28



# Search Requested

EXAMINER: JOSEPH D. ANTHONY

REM 10D21

(571) 272-1117

ART UNIT 1714

SEARCH SUBMITTED: 02/02/04

APPLICATION: 09/786,719

FILED: 8/2/99

PAPER  
SEARCH  
REQUESTED

- A) Please <sup>STRUCTURE</sup> SEARCH the complex compound of CHIM 1.
- 2) Please also search where in the complex compound of CHIM 1 is microencapsulated in a polymer shell (e.g. polyethylene, polyorganosiloxanes)
- 3) Please also search a process of using the complex compound of CHIM 1 by adding it to polymers as a FLAME or FIX or COMBUSTION RETARDANT.

- COPY OF pending CHIM attached
- COPY OF 371 404 Report enclosed

**ABSTRACT**

A description is given of a combustion retardant for polymer materials in the form of a new complex compound of the ammonium salt of amide of  
5 alkylphosphonic acid with ammonium chloride, and also of processes for producing various polymer materials using the said combustion retardant.

09786749.062201



APPLICANTS: Zubkova et al

DATE: June 18, 2001

SERIAL NO.: 09/786,719

GROUP ART UNIT:

INTERNATIONAL FILING DATE: 02 August 1999

EXAMINER:

FOR: "COMBUSTION RETARDANT FOR POLYMERIC MATERIALS"

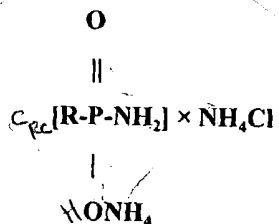
ATTORNEY DOCKET NO.: A01058US (98148.13)

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Copy of claims showing changes

Set out below is a copy of claims showing changes made in this amendment, with added matter underlined and omitted matter in brackets:

-- 1. Complex compound of ammonia salt of amide of alkylphosphonic acid with ammonium chloride of formula (I)



where R is the alkyl radical C-1-3.

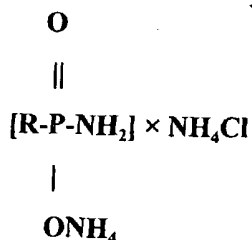
• 2 NH<sub>3</sub>  
• NH<sub>4</sub> Cl  
• HCl

2. Complex compound in accordance with Claim 1, in which there are about 1.8 molecules of ammonium chloride to one molecule of ammonia salt of amide of alkylphosphonic acid.

3. Process for producing a complex compound of ammonia salt of amide of alkylphosphonic acid with ammonium chloride of Formula (I), consisting of the interaction of dichloroanhydride of alkylphosphonic acid with gaseous ammonia in a medium of organic solvent at temperature 10-20°C.

4. Combustion retardant for polymer materials, consisting of a complex compound of ammonia salt of amide of alkylphosphonic acid with ammonium chloride of formula (I)

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where R is the alkyl radical C-1-3.

5. Combustion retardant in accordance with Claim 4, in which there are about 1.8 molecules of ammonium chloride to one molecule of ammonia salt of amide of alkylphosphonic acid.

6. (Amended) Combustion retardant in accordance with Claim 4 [or 5], *characterised* in that it is microencapsulated in a polymer shell.

7. Combustion retardant in accordance with Claim 6, *characterised* in that the said polymer shell is made of polyethylene.

8. Combustion retardant in accordance with Claim 6, *characterised* in that the said polymer shell is made of polyorganosiloxanes.

9. Combustion retardant in accordance with Claim 8, *characterised* in that the polyorganosiloxanes are selected from a group including polyvinylmethyldiethoxy-siloxane and polyaminopropylethoxysiloxane.

10. Process for producing low fire risk polymer materials by the introduction of the CR into the polymer in the course of its processing, *characterised* in that the CR used is a complex compound of ammonia salt of amide of alkylphosphonic acid with ammonium chloride of Formula (I).

11. Process for producing low fire risk polymer materials in accordance with Claim 10, *characterised* in that it includes the following sequence of operations:

- joint extrusion of the said combustion retardant with the polymer;
- moulding the polymer fibre;
- granulation.

12. Process for producing low fire risk polymer materials in accordance with Claim 10, *characterised* in that it includes the following sequence of operations:

- mixing of the said combustion retardant with the polymer composition;
- rolling the mass;
- pressing the articles.

13. (Amended) Process in accordance with [any of] Claim[s] 10[-12], *characterised* in that the combustion retardant is first microencapsulated in a polymer shell.

14. Process in accordance with Claim 13, *characterised* in that the size of the microcapsules is 5-25  $\mu$ m.

15. Process in accordance with Claim 13, *characterised* in that the polymer shell is made of polyethylene with shell content including 10-15 wt.% of combustion retardant.

16. Process in accordance with Claim 13, *characterised* in that polyorganosiloxanes are used for the polymer shell.

17. Process in accordance with Claim 16, *characterised* in that the polyorganosiloxane consists of polymethyldiethoxysiloxanes with shell containing 2-5 wt.% of combustion retardant.

18. Process in accordance with Claim 16, *characterised* in that the polyorganosiloxane used is polyaminopropylethoxysiloxane, with shell containing 2-5 wt.% of combustion retardant.

19. (Amended) Process in accordance with [any of] Claim[s] 10[-18], *characterised* in that polyethylene, polypropylene and copolymers of various compositions based on them are processed.



20. (Amended) Process in accordance with [any of] Claim[s] 10[-18], *characterised* in that polystyrene and copolymers of various compositions based on it are processed.

21. Process for producing low fire risk polymer materials by the introduction of combustion retardant into the polymer, *characterised* in that the combustion retardant used is a complex compound of ammonia salt of amide of alkylphosphonic acid with ammonium chloride of Formula (I), which is introduced into the polymer composition before it sets.

22. Process in accordance with Claim 21, *characterised* in that a filler is introduced into the polymer composition along with the said combustion retardant, and as a result of the saturation of the filler with the setting polymer composition, low fire risk materials are produced.

23. (Amended) Process in accordance with Claim 21[ or 22], *characterised* in that polyesters are processed.

24. (Amended) Process in accordance with Claim 21[ or 22], *characterised* in that epoxy resins are processed.

25. Process for producing low fire risk polymer materials, *characterised* in that a complex compound of ammonia salt of amide of alkylphosphonic acid with ammonium chloride of Formula (I) is introduced into a polymer composition including synthetic rubber, after which it is rolled before the article is pressed.

26. Low fire risk polyethylene produced by a process in accordance with Claim 6.

27. Low fire risk polypropylene produced by a process in accordance with Claim 6.

28. Low fire risk polystyrene produced by a process in accordance with Claim 6.

29. Low fire risk copolymers based on polyethylene, polypropylene and polystyrene,

produced by a process in accordance with Claim 6.

30. Low fire risk polyesters produced by a process in accordance with Claim 21.

31. Low fire risk epoxy resins produced by a process in accordance with Claim 21.

32. Low fire risk composition materials produced by a process in accordance with Claim 22.

33. Low fire risk synthetic rubbers produced by a process in accordance with Claim 25.

34. Low fire risk polycapraamide materials produced by a process in accordance with Claim 13.

35. Low fire risk polymethyl methacrylate compositions produced by a process in accordance with Claim 11. --

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35. Low fire risk polymethyl methacrylate compositions produced by a process in accordance with Claim 11.--

**REMARKS**

The claims have been amended to reduce claims fees. For convenience in prosecution, all claims are repeated herein, even those which are not amended herein. Two sets of claims are included, one set (attached) showing the changes made in this response and one clean set (set out above). No new matter has been added to the application.

Enclosed are the following:

1. Declaration;
2. Power Of Attorney by the Assignee;
3. Statement under 37 C.F.R. § 3.73(b);
4. Copy of Assignment (The assignment has been requested to be recorded with the Patent and Trademark Office);
5. \$255.00 for Excess Claim Fees (Applicant qualifies for small entity status); and
6. Copy of Formalities letter;

Applicants have not included the Oath Surcharge under 37 C.F.R. §1.492(e) because this surcharge was paid with the original papers entering the national stage. If this is incorrect, the office is authorized to charge said surcharge to Deposit Account Number 50-0694.

Applicants respectfully submit that the application is in condition for allowance. A Notice of Allowance is hereby respectfully requested.

Should the Examiner feel that a telephone conference would advance the prosecution of this application, he is encouraged to contact the undersigned at the telephone number listed below.

Applicants respectfully petition the Commissioner for any extension of time necessary to render this paper timely.

09786719-062201

09/736 719  
Translation  
5060

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

10

Applicant's or agent's file reference	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/RU99/00273	International filing date (day/month/year) : 02 August 1999 (02.08.99)	Priority date (day/month/year) 08 September 1998 (08.09.98)
International Patent Classification (IPC) or national classification and IPC C07F 9/44, C08K 5/5399, 9/10, C09K 21/14, C08J 5/00, 5/24		
Applicant ISLE FIRESTOP LIMITED		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.
2. This REPORT consists of a total of <u>3</u> sheets, including this cover sheet.  <input type="checkbox"/> This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).  These annexes consist of a total of _____ sheets.
3. This report contains indications relating to the following items:  I <input checked="" type="checkbox"/> Basis of the report II <input type="checkbox"/> Priority III <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability IV <input type="checkbox"/> Lack of unity of invention V <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement VI <input type="checkbox"/> Certain documents cited VII <input type="checkbox"/> Certain defects in the international application VIII <input type="checkbox"/> Certain observations on the international application

Date of submission of the demand 07 April 2000 (07.04.00)	Date of completion of this report 11 August 2000 (11.08.2000)
Name and mailing address of the IPEA/RU	Authorized officer
Facsimile No.	Telephone No.

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.  
PCT/RU 99/00273

## V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

## 1. Statement

Novelty (N)	Claims	1-35	YES
	Claims		NO
Inventive step (IS)	Claims	1-35	YES
	Claims		NO
Industrial applicability (IA)	Claims	1-35	YES
	Claims		NO

## 2. Citations and explanations

In drawing up this report, the following documents were taken into account:

D1: EP-A-0 254 683; D3: RU-A-2 024 560;  
D2: US-A-4 308 197; D4: WO-A-87/00187;  
D5: Encyclopaedia of Polymers, Moscow, Soviet  
Encyclopaedia, 1974, Vol.2, pages 247-248;  
D6: RU-A-2 103 314;  
D7: GB-A-1 526 361;  
D8: RU-A-2 099 367.

Claim 1 claims a complex compound of ammonium salt of alkylphosphonic acid amide with ammonium chloride of formula (1).

The known prior art closest to the invention according to Claim 1 is represented by D1.

D1 describes a dimethyl, monomethylphosphonate or methylphosphonic acid salt with a compound of formula (1).

The invention according to Claim 1 differs from D1 in terms of a different type of phosphoro-organic salt.

Documents D2 to D8 do not disclose a complex compound, as claimed in Claim 1 of the present application.

The invention according to Claims 1 and 2 therefore meets the criteria of novelty and inventive step.

Claim 3 claims a method of producing a complex compound according to Claim 1.

The closest prior art method of those known is that described in D2. The known method of obtaining ammonia salt of alkylphosphonic acid consists in producing a reaction between alkylphosphonic acid and gaseous ammonia in an organic solvent medium at a temperature of between - 60°C and - 70°C.

The invention according to Claim 3 differs from an organic salt with different chemical properties owing to the use of a different starting substance and different temperature conditions for the reaction between the components.

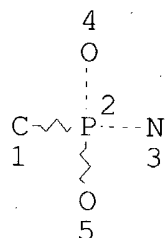
The invention according to Claim 3 therefore meets the criteria of novelty and inventive step.

Claim 4 claims a combustion retardant for polymeric materials, which is a complex compound according to Claim 1. Taking into account the above analysis, carried out in relation to Claim 1, it should be concluded that Claims 4 to 9 meet the criteria of novelty and inventive step.

Since Claims 10 to 35 relate to the invention claimed in Claim 1, they also meet the criteria of novelty and inventive step.

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L5 STR



## NODE ATTRIBUTES:

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NSPEC   IS RC      AT      1
CONNECT IS E1   RC AT      3
CONNECT IS E1   RC AT      4
CONNECT IS E1   RC AT      5
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

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## GRAPH ATTRIBUTES:

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RING(S) ARE ISOLATED OR EMBEDDED
NUMBER OF NODES IS      5

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STEREO ATTRIBUTES: NONE

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      STRUCTURE

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L3  STRUCTURE
L4  10 S L3 SSS SAM

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L5  FILE 'LREGISTRY' ENTERED AT 11:40:28 ON 04 FEB 2004
      STRUCTURE
L6  0 S L5 SSS SAM
L7  0 S L3 SSS SAM

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L8  FILE 'REGISTRY' ENTERED AT 11:45:16 ON 04 FEB 2004
      4 S L5 SSS SAM
L9  10 S L3 SSS SAM
L10 4 S L5 SSS SAM

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FILE 'HCAPLUS' ENTERED AT 11:53:16 ON 04 FEB 2004  
E ZUBKOVA/AU  
E ZUBKOVA N?/AU  
L11 52 S E1 OR E7 OR E8  
E BUTYLKINA N?/AU  
L12 22 S E2 OR E4 OR E5  
L13 16 S L11 AND L12  
E KHALTURINSKY N?/AU  
E KHALTURINSK? N?/AU  
L14 137 S E4-E12  
L15 6 S L13 AND L14  
E BERLIN A?/AU  
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L16 1643 S E4 OR E23 OR E26-E31  
L17 3 S L15 AND L16

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SELECT L17 RN 1-3

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FILE 'LREGISTRY' ENTERED AT 12:05:44 ON 04 FEB 2004

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L19 4 S L5 SSS SAM  
L20 118 S L5 SSS FULL  
SAVE L20 ANT719/A

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L21 43 S L20

FILE 'REGISTRY' ENTERED AT 13:39:35 ON 04 FEB 2004  
E PHOSPHONAMIDIC ACID/CN  
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L22 1 S E4  
L23 7 S METHYL(W) PHOSPHONAMIDIC(W) ACID  
E PHOSPHONAMIDIC ACID/CN



E PHOSPHORIC DICHLORIDE/CN  
 E AMMONIA/CN  
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 L30 3498 S (PHOSPHONAMIDIC)  
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 L34 0 S ETHYLPHOSPHONAMIDIC (W) ACID  
 L35 1 S ETHYLPHOSPHONAMIDIC

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FILE 'REGISTRY' ENTERED AT 14:05:01 ON 04 FEB 2004

E AMIDOPHOSPHONIC ACID/CN  
 L36 0 S E3 AND ETHYL  
 L37 1608 S (PHOSPHONAMIDIC (W) ACID) AND ETHYL  
 L38 2 S (PHOSPHONAMIDIC (W) ACID) (W) ETHYL  
 L39 67 S (PHOSPHONAMIDIC (W) ACID) (W) P (W) ETHYL

FILE 'HCAPLUS' ENTERED AT 14:09:01 ON 04 FEB 2004

L40 393 S (14500-78-8/RN OR 52030-74-7/RN OR ?PHOSPHONAMIDIC?)  
 L41 2628 S (676-97-1/RN OR 1066-50-8/RN OR 4708-04-7/RN OR 1498-46  
 L42 411808 S 7664-41-7/RN OR 1336-21-6/RN OR NH3 OR NH4OH OR AMMONIA  
 L43 21 S L40 AND L42  
 L44 78 S L41 AND L42  
 L45 94 S L43 OR L44  
 L46 38995 S (FIRE? OR FLAME OR COMBUST? OR DECOMPOSIT? OR PYROLY?) (   
 L47 39506 S FIREPROOF? OR FLAMEPROOF? OR FIRERETARDANT? OR FLAMERET  
 L48 59503 S L46 OR L47  
 L49 2964 S L40 OR L41  
 L50 214 S L49 AND L48  
 L51 15 S L45 AND L48  
 L52 15 S L51 NOT L21

L53 15 S L50 AND L42  
 L54 0 S L53 NOT (L21 OR L52)  
 L55 191 S L50 AND ((1907-1999)/PY OR (1907-1999)/PRY)  
 L56 151 S L55 AND PATENT/DT  
 L57 153079 S L42/TI  
 L58 5 S L50 AND L57  
 L59 0 S L58 NOT (L21 OR L52)  
 L60 137690 S L42/ABS  
 L61 399025 S L42/AB  
 L62 14 S L55 AND L61  
 L63 0 S L62 NOT (L21 OR L52)

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L15 ANSWER 1 OF 6 HCAPLUS COPYRIGHT 2004 ACS on STN  
 2001:78393 Document No. 134:148350 Method for producing polymer materials with reduced fire risk. **Zubkova, Nina Sergeevna; Butylkina, Nataliya Grigorievna; Khalturinsky, Nikolai Alexandrovich**; Berlin, Alexandr Alexandrovich (Isle Firestop Limited, UK). PCT Int. Appl. WO 2001007448 A1 20010201, 18 pp. DESIGNATED STATES: W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM; RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE. (Russian). CODEN: PIXXD2. APPLICATION: WO 1999-RU255 19990726.

AB A combustion retardant for polymer materials consists of a methacrylate of the formula  $\text{CH}_2:\text{CMeCO}_2\text{CH}_2\text{CH}_2\text{OPMe(O)(OR)(I)}$ , where R is C1-4 alkyl. I can be used as a comonomer by copolymn. with Me methacrylate and also for imparting fire resistance to chem. fibers. In the last case, cotton, rayon, or polyamide fibers are graft polyimd. with I.

IC ICM C07F009-40  
 ICS C08F230-02; C08F220-14; C08F220-18; D06M014-04; D06M014-16; C09K021-14

CC 37-3 (Plastics Manufacture and Processing)  
 Section cross-reference(s): 40

L15 ANSWER 2 OF 6 HCAPLUS COPYRIGHT 2004 ACS on STN  
 X 2000:862776 Document No. 134:179280 Characteristics of carbonization of polycapraamide in the presence of combustion retardants containing phosphorus and silicon. **Kuznetsova, O. G.; Zubkova, N. S.; Butylkina, N. G.; Naganovskii, Yu. K.; Dubnikova, I. L.; Khalturinskii, N. A.** (Mosk. Gos. Tekstil'naya Akad., Moscow, Russia). Plasticheskie Massy (10), 19-21 (Russian) 2000. CODEN: PLMSAI. ISSN: 0554-2901. Publisher:

ZAO NP "Plasticheskie Massy".

AB Poly(ethoxyvinylsiloxane) and poly[(3-aminopropyl)ethoxysiloxane] were used as encapsulating materials for T 2 fireproofing agent incorporated into polycapraamide during the press-forming. Microencapsulation of the fireproofing agent resulted in the oxygen index and carbonization rate raise. Introduction of microencapsulated T 2 decreased polycapraamide glass transition without affecting melting temp.

CC 37-6 (Plastics Manufacture and Processing)

L15 ANSWER 3 OF 6 HCAPLUS COPYRIGHT 2004 ACS on STN

2000:175862 Document No. 132:208728 Fire retardant for polymeric materials and its use. **Zubkova, Nina Sergeevna;**

**Butylkina, Nataliya Grigorievna; Khalturinsky, Nikolai Alexandrovich;** Berlin, Alexandr Alexandrovich; Vilesova, Marina Sergeevna; Bosenko, Margarita Serafimovna; Voronkova, Ljudmila Ivanovna (Isle Firestop Ltd., UK). PCT Int. Appl. WO 2000014152 A1 20000316, 22 pp. DESIGNATED STATES: W: AL, AM, AU, BB, BR, CA, CN, CZ, EE, FI, GE, HU, IS, JP, KE, KG, KP, KR, LK, LR, LS, LT, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, RO, RU, SD, SG, SI, SK, TR, TT, UA, UG, US, UZ, VN, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM; RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE. (Russian). CODEN: PIXXD2. APPLICATION: WO 1998-RU289 19980908.

AB The fire retardant consists of a complex comprising an ammonium salt of a Cl-3-alkylphosphonamide as well as NH4Cl. Methods for producing safer forms of various polymeric materials using this fire retardant, preferably in microencapsulated form, are also provided.

IC ICM C08K005-5399

ICS C08K009-10; C09K021-14; C08L009-00; C08L011-00; C08L013-00; C08L019-00; C08L021-00; C08L023-04; C08L023-10; C08L025-04; C08L063-00; C08L067-00; C08J005-24; C07F009-44

CC 37-6 (Plastics Manufacture and Processing)

L15 ANSWER 4 OF 6 HCAPLUS COPYRIGHT 2004 ACS on STN

1999:529316 Document No. 131:171523 Fireproofing textile materials.

*Good but not prior art*

**Zubkova, Nina Sergeevna; Butylkina, Nataliya Grigorievna;** Berlin, Alexandr Alexandrovich; **Khalturinsky, Nikolai Alexandrovich;** Galbraikh, Leonid Semenovich (Isle Firestop Ltd., UK). PCT Int. Appl. WO 9941446 A1 19990819, 18 pp. DESIGNATED STATES: W: AL, AM, AU, BB, BG, BR, CA, CN, CZ, EE, FI, GE, HU, IS, JP, KE, KG, KP, KR, LK, LR, LS, LT, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, RO, SD, SG, SI, SK, TR, TT, UA, UG, US, UZ, VN, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM; RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE. (Russian). CODEN: PIXXD2. APPLICATION: WO 1998-RU31 19980213.

AB Textile materials are 1st impregnated with a melamine-type resin, e.g., metazin, or with organosilicone-contg. substances, e.g., (EtO)3SiCH2CH2CH2NH2. The materials are dried, then further

impregnated with aq. soln. contg. urea and a complex salt  $\text{RP(O)(ONH}_4\text{)NH}_2 \cdot \text{NH}_4\text{Cl}$  ( $\text{R} = \text{C1-3 alkyl}$ ) obtained from amidoalkylphosphonic acid and  $\text{NH}_4\text{Cl}$  (no data), and dried at 140-170.degree.. The materials processed according to this method, esp. fabrics made of cotton fiber mixts. with synthetic fibers contg. >20% of the latter fibers, have a reduced combustibility while the gases produced during combustion have a low toxicity.

IC ICM D06M013-364  
ICS D06M013-432; D06M013-447  
CC 40-9 (Textiles and Fibers)

L15 ANSWER 5 OF 6 HCAPLUS COPYRIGHT 2004 ACS on STN

1998:663560 Document No. 129:343993 Rheological and fireproofing characteristics of polyethylene modified with a microencapsulated fire retardant. Zubkova, N. S.; Butylkina, N. G.; Chekanova, S. E.; Tyuganova, M. A.; Khalturinskii, N. A.; Reshetnikov, I. S.; Naganovskii, Yu. K. (Institute of Synthetic Polymer Materials, Moscow State Textile Academy, Russian Academy of Sciences, Russia). Fibre Chemistry (Translation of Khimicheskie Volokna), 30(1), 11-13 (English) 1998. CODEN: FICYAP. ISSN: 0015-0541. Publisher: Consultants Bureau.

AB It was found that incorporation of the microencapsulated fireproofing compd. (MIC FC) T-2 in silicon-contg. shells in polyethylene does not alter the effective viscosity of the polymer melt at 200.degree.C. During thermolysis of PE contg. MIC FC in a poly(vinylethoxysiloxane) (PVES) shell, the max. rate of liberation of volatile compds. is 1.8 times lower than for the starting PE and is shifted to the region of a higher degree of conversion of the polymer, the amt. of gaseous compds. decreases, and the yield of carbonized residue (CR) increases. The fire hazard of PE modified by MIC FC T-2 in a PVES shell decreases to a greater degree than when MIC FC in a PE shell is used.

CC 37-5 (Plastics Manufacture and Processing)

L15 ANSWER 6 OF 6 HCAPLUS COPYRIGHT 2004 ACS on STN

1997:717023 Document No. 128:4393 Fireproofing polyethylene and polypropylene. Zubkova, N. S.; Tyuganova, M. A.; Butylkina, N. G.; Khalturinskii, N. A.; Reshetnikov, I. S.; Potapova, E. V.; Vilesova, M. S.; Voronkova, L. I.; Bosenko, M. S. (Russia). Plasticheskie Massy (5), 35-37 (Russian) 1996. CODEN: PLMSAI. ISSN: 0554-2901. Publisher: NPAOZT "Plastmassy".

AB Thermal properties of fireproofing compns. of polyethylene and polypropylene contg. Antipyrène T-2 microencapsulated in polyethylene and polyvinyltriethoxysilane were studied. Thermal effects of thermal decompn. and thermophys. characteristics near m.p. were detd.

CC 38-3 (Plastics Fabrication and Uses)

Note: Antipyrène is a technical mixture of two individual compounds - 1) the ammonium salt of methyldiphosphonic acid and 2) ammonium cyanide. This is not a complex as claimed by applicants claim 1. (See Applicant's specification on page 8.)

=> d 121 1 13 14 15 26 cbib abs hitstr hitind

L21 ANSWER 1 OF 43 HCAPLUS COPYRIGHT 2004 ACS on STN

2003:76792 Document No. 138:138107 Production of ammonium salt of nitrilotris(methylenephosphonic acid amide) and its use as flame retardant. Zubkova, Nina Sergeevna; Butylkina, Nataliya Grigorievna (Russia). PCT Int. Appl. WO 2003008426 A1 20030130, 21 pp.

DESIGNATED STATES: W: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM; RW: AT, BE, BF, BJ, CF, CG, CH, CI, CM, CY, DE, DK, ES, FI, FR, GA, GB, GR, IE, IT, LU, MC, ML, MR, NE, NL, PT, SE, SN, TD, TG, TR. (Russian). CODEN: PIXXD2. APPLICATION: WO 2001-RU293 20010716.

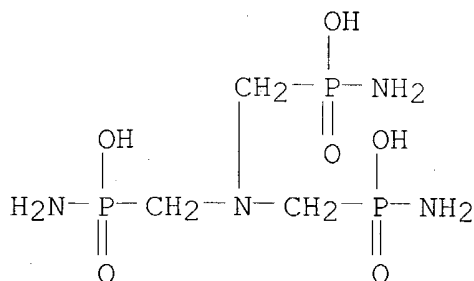
AB Ammonium salt of nitrilotris(methylenephosphonic acid amide) is produced by reacting nitrilotris(methylenephosphonic acid) and urea at the ratio of 1:4 by slow raising of the reaction temp. from 100 to 200.degree. and heating the mixt. melt for 12 h. The compd. is used as flame retardant for plastics, textiles and wood. Thus, ammonium salt of nitrilotris(methylenephosphonic acid amide) was produced and used as flame retardant for polypropylene by mixing with polypropylene crumb and extruding the compn. at 220.degree..

IT **491870-41-8P**

(prodn. of ammonium salt of nitrilotris(methylenephosphonic acid amide) and its use as flame retardant)

RN 491870-41-8 HCAPLUS

CN Phosphonamidic acid, P,P',P''-[nitrilotris(methylene)]tris-, triammonium salt (9CI) (CA INDEX NAME)



IC ICM C07F009-44  
ICS C08K005-5399; C09K021-12; C09K021-14; C08L077-02; C08L023-06;  
C08L023-12; D06M013-447; B27K003-36  
CC 37-6 (Plastics Manufacture and Processing)  
Section cross-reference(s): 40, 43  
IT **491870-41-8P**  
(prodn. of ammonium salt of nitrilotris(methylenephosphonic acid  
amide) and its use as flame retardant)

L21 ANSWER 13 OF 43 HCAPLUS COPYRIGHT 2004 ACS on STN  
1998:315697 Document No. 128:295628 Fire-protected thermoplastic  
polymer composition containing encapsulated ammonium  
methylphosphonamide. Zubkova, Nina S.; Tyuganova, Margarita A.;  
Moryganov, Andrej P.; Borovkov, Nikolaj Yu (Moskovskaya  
Gosudarstvennaya Tekstilnaya Akademiya Im.A.N.Kosygina, Russia).  
Russ. RU 2099384 C1 19971220 From: Izobreteniya 1997, (35), 473.  
(Russian). CODEN: RUXXE7. APPLICATION: RU 1995-122538 19951229.

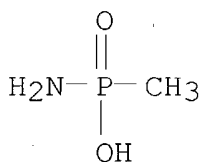
AB Title only translated.

IT **106912-93-0**  
(fire-protected thermoplastic polymer compn. contg. encapsulated  
ammonium methylphosphonamide)

RN 106912-93-0 HCAPLUS

CN Phosphonamidic acid, P-methyl-, monoammonium salt (9CI) (CA INDEX  
NAME)

*GOOD CITE, BUT NOT A COMPLEX  
WITH AMMONIUM CHLORIDE*



● NH<sub>3</sub>

IC ICM C09K021-12  
ICS C09K021-14; C08K005-5399; C08K009-10  
CC 38-3 (Plastics Fabrication and Uses)  
IT **106912-93-0**  
(fire-protected thermoplastic polymer compn. contg. encapsulated  
ammonium methylphosphonamide)

L21 ANSWER 14 OF 43 HCAPLUS COPYRIGHT 2004 ACS on STN  
1998:51091 Document No. 128:89681 Decreasing the combustibility of  
polyolefins using microencapsulated fire retardants. Zubkova, N.

S.; Tyuganova, M. A.; Reshetnikov, I. S.; Khalturinskii, N. A. (Moscow State Textile Academy, Institute of Synthetic Polymer Materials, Russian Academy of Sciences, Russia). Fibre Chemistry (Translation of Khimicheskie Volokna), Volume Date 1997, 29(3), 166-168 (English) 1998. CODEN: FICYAP. ISSN: 0015-0541. Publisher: Consultants Bureau.

AB A method of decreasing the combustibility of polyolefins by incorporation of microencapsulated fire retardants (ME FR) in the polymer melt was developed. PE and PP contg. ME FR in PE and polyvinyltriethoxysilane (PVTES) shells belong to the category of difficult to burn materials. The study of the thermophys. and physicochem. characteristics of modified PE and PP demonstrated the major possibility of realization of the method for fabrication of polyolefins with a low fire hazard.

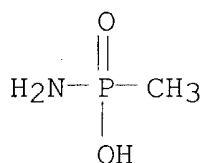
IT 106912-93-0

(microencapsulated; decreasing the combustibility of polyolefins using microencapsulated fire retardants)

RN 106912-93-0 HCAPLUS

CN Phosphonamidic acid, P-methyl-, monoammonium salt (9CI) (CA INDEX NAME)

*Good BUT NOT A complex  
with AMMONIUM chloride*



● NH<sub>3</sub>

CC 38-3 (Plastics Fabrication and Uses)

IT 106912-93-0

(microencapsulated; decreasing the combustibility of polyolefins using microencapsulated fire retardants)

L21 ANSWER 15 OF 43 HCAPLUS COPYRIGHT 2004 ACS on STN

1997:763503 Document No. 128:62773 Thermal decomposition and combustion of phosphorus-and metal-containing cellulosic materials. Seredina, M. A.; Tyuganova, M. A. (Russia). Khimicheskie Volokna (5), 38-40 (Russian) 1995. CODEN: KVLKA4. ISSN: 0023-1118. Publisher: Khimicheskie Volokna.

AB Fireproofing compns. contg. ammonium salt of P-methylphosphonoamidic acid and copper, cobalt, chromium, or zinc sulfate showed high efficiency in fireproofing cotton and rayon fabrics. The presence of these fireproofing agents changed the course of thermodegrdn. of

the cellulosic materials and decreased significantly the amt. of toxic pyrolysis products.

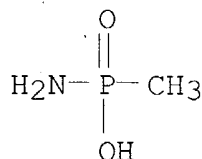
IT 106912-93-0

(thermal decompn. and combustion of cellulosic fabrics fireproofed with phosphorus-and metal-contg. agents)

RN 106912-93-0 HCAPLUS

CN Phosphonamidic acid, P-methyl-, monoammonium salt (9CI) (CA INDEX NAME)

*good but not a complex with  
ammonium chloride*



● NH<sub>3</sub>

CC 40-9 (Textiles and Fibers)

IT 7733-02-0, Zinc sulfate 7758-98-7, Copper sulfate, uses  
10124-43-3, Cobalt sulfate 14489-25-9, Chromium sulfate  
106912-93-0

(thermal decompn. and combustion of cellulosic fabrics fireproofed with phosphorus-and metal-contg. agents)

✓ L21 ANSWER 26 OF 43 HCAPLUS COPYRIGHT 2004 ACS on STN

1987:86418 Document No. 106:86418 Inhibition of the combustion of cellulose by phosphorus-containing compounds. 5. Relation between super-equilibrium flame ionization and fire resistance. Sultanov, M T.; Sadykov, M. M.; Muratova, U. M.; Sultanov, F. Kh.; Tashpulatov, Yu. T.; Usmanov, Kh. U. (NII Khim. Tekhnol. Khlopkovoi Tsellyul., USSR). Koksnes Kimija (6), 44-6 (Russian) 1986. CODEN: KHDRDQ. ISSN: 0201-7474.

AB Study of the inhibition of combustion of cellulose (I) [9004-34-6] by neutralization of charges in the flame found during burning of I via addn. of P-contg. compds. showed that the contribution of super-equil. ionization to combustion inhibition of I was negligible. For this mechanism to be activated, very high concns. of P-contg. compds. in I would be required [e.g., 12% P(CH<sub>2</sub>OH)<sub>3</sub> or 40%, H<sub>3</sub>PO<sub>4</sub>], whereas sufficient fireproofing of I can be obtained on addn. of 1.0% P(CH<sub>2</sub>OH)<sub>3</sub> or 1.7% H<sub>3</sub>PO<sub>4</sub>.

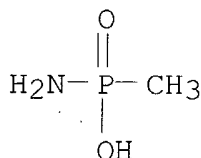
IT 106912-93-0

(fireproofing by, of cellulose, super-equil. flame ionization in relation to)

RN 106912-93-0 HCAPLUS



CN Phosphonamidic acid, P-methyl-, monoammonium salt (9CI) (CA INDEX NAME)



*Good but not a complex  
with Ammonia chloride*

● NH<sub>3</sub>

CC 43-3 (Cellulose, Lignin, Paper, and Other Wood Products)  
IT 993-13-5, Methyl phosphonic acid 2767-80-8,  
Tris(hydroxymethyl)phosphine 4759-30-2, Methyl phosphonic diamide  
7664-38-2, Phosphoric acid, uses and miscellaneous 7783-28-0,  
Diammonium phosphate 27546-19-6 **106912-93-0**  
(fireproofing by, of cellulose, super-equil. flame ionization in  
relation to)

X => d 152 1-15 cbib abs hitstr hitind

L52 ANSWER 1 OF 15 HCAPLUS COPYRIGHT 2004 ACS on STN  
2002:709675 Document No. 137:217752 Phosphorous-containing  
multifunctional compounds as crosslinking agent imparting  
**flame retardancy**. Guo, Bing-Lin; Wang,  
Jia-Shiang; Chen, Bo-Chiuan; Hung, Kuen-Ming (Taiwan). Taiwan TW  
401433 B 20000811, 21 pp. (Chinese). CODEN: TWXXA5. APPLICATION:  
TW 1998-87119530 19981123.

AB This phosphoramidate or phosphonamide compds.  
AR<sub>2</sub>(NHP(O)R<sub>1</sub>NHR<sub>2</sub>)<sub>a</sub>NHP(O)R<sub>1</sub>NHR<sub>2</sub>A are prepd.; A = NH<sub>2</sub>, H or none (R<sub>2</sub> =  
H); R<sub>1</sub> = OR, Ar, OAr, NHR, NH(CH<sub>2</sub>)<sub>n</sub>Ar; R = satd. or unsatd. aliph.  
group; Ar = arom. group; R<sub>2</sub> = H, alkylenediamine residue, n = 1-30;  
a = 0-1000; X = CH<sub>2</sub>, O, SO<sub>2</sub>. These compds. can be used to prep.  
polyurethane or epoxy resin with good fire resistance. Reaction of  
POCl<sub>3</sub> with n-BuNH<sub>2</sub> in PhMe gave N,N',N''-tributylphosphoramidate,  
useful as crosslinking agent of epoxy resins.

IT **7664-41-7, Ammonia**, reactions  
(phosphorous-contg. multifunctional compds. as crosslinking agent  
imparting **flame retardancy**)

RN 7664-41-7 HCAPLUS

CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH3

- IC ICM C08G079-02  
 CC 37-6 (Plastics Manufacture and Processing)  
 Section cross-reference(s): 42  
 ST phosphoramidate phosphonamide crosslinking agent **flame retardancy**; butylamine phosphoryl chloride  
 tributylphosphoramidate crosslinker  
 IT Coating materials  
 (fire-resistant; phosphorous-contg. multifunctional compds. as crosslinking agent imparting **flame retardancy**)  
 IT Crosslinking agents  
 Fire-resistant materials  
**Fireproofing agents**  
 (phosphorous-contg. multifunctional compds. as crosslinking agent imparting **flame retardancy**)  
 IT Epoxy resins, preparation  
 (phosphorous-contg. multifunctional compds. as crosslinking agent imparting **flame retardancy**)  
 IT Polyurethanes, preparation  
 (polyoxyalkylene-; phosphorous-contg. multifunctional compds. as crosslinking agent imparting **flame retardancy**)  
 IT 106-92-3DP, Allyl glycidyl ether, reaction product with H siloxane 161127-41-9DP, Methylhydrogensilanediol-octamethylcyclotetrasiloxane copolymer, reaction product with allyl glycidyl ether  
 (phosphorous-contg. multifunctional compds. as crosslinking agent imparting **flame retardancy**)  
 IT 4707-88-4P; Phenylphosphonic diamide 14360-81-7P 19622-52-7P 20638-28-2P 23344-69-6P 25190-24-3P 25190-26-5P 25279-96-3P, Hexamethylenediamine-**phenylphosphonic dichloride** copolymer 25279-98-5P, p-Phenylenediamine-**phenylphosphonic dichloride** copolymer 25949-16-0P, Ethylenediamine-**phenylphosphonic dichloride** copolymer, sru 28851-33-4P, **Phenylphosphonic dichloride** -p-diaminodiphenylmethane copolymer 28851-34-5P, Ethylenediamine-**phenylphosphonic dichloride** copolymer 31868-37-8P, **Phenylphosphonic dichloride** -p-diaminodiphenylmethane copolymer, sru 37624-66-1P 53721-41-8P 99190-25-7P 453537-97-8P 453557-97-6P  
 (phosphorous-contg. multifunctional compds. as crosslinking agent imparting **flame retardancy**)  
 IT 23344-69-6DP, reaction product with siloxane glycidyl ether 37624-66-1DP, reaction product with siloxane glycidyl ether 319915-23-6DP, EP 128, reaction product with phosphoramides and siloxane 453537-98-9P 453537-99-0P 453538-00-6P 453538-01-7P

453538-02-8P

(phosphorous-contg. multifunctional compds. as crosslinking agent  
imparting **flame retardancy**)

- IT 100-46-9, Benzylamine, reactions 101-77-9 107-10-8,  
n-Propylamine, reactions 107-15-3, Ethylenediamine, reactions  
108-95-2, Phenol, reactions 109-73-9, Butylamine, reactions  
124-09-4, Hexamethylenediamine, reactions 141-43-5, Ethanolamine,  
reactions 824-72-6, **Phenylphosphonic dichloride**  
2240-41-7, Dimethyl phenylphosphonate **7664-41-7**,  
**Ammonia**, reactions 10025-87-3, Phosphoryl chloride  
20638-26-0 25265-76-3, Phenylenediamine  
(phosphorous-contg. multifunctional compds. as crosslinking agent  
imparting **flame retardancy**)

L52 ANSWER 2 OF 15 HCAPLUS COPYRIGHT 2004 ACS on STN

X/2001:670347 Document No. 135:372458 **Flame-retarding**  
materials, 3: tailor-made thermal stability epoxy curing agents  
containing difunctional phosphoric amide groups. Kuo, Ping-Lin;  
Wang, Ja-Shung; Chen, Po-Chuan; Chen, Leo-Wang (Department of  
Chemical Engineering, National Cheng Kung University, Tainan, 70101,  
Taiwan). Macromolecular Chemistry and Physics, 202(11), 2175-2180  
(English) 2001. CODEN: MCHPES. ISSN: 1022-1352. Publisher:  
Wiley-VCH Verlag GmbH.

AB Four different phosphorus amides, p-phenylphosphonic diamide  
[PhPO(NH<sub>2</sub>)<sub>2</sub>], phenylphosphonic bis(N-butylamide) (2-BU-PH),  
phenylphosphoric bis(N-butylamide) (2-BU-PHO), and phenylphosphonic  
bis(N-benzylamide) (2-BZ-PH), have been successfully synthesized by  
low temp. condensation and characterized by FT-IR and 1H NMR and 31P  
NMR spectra. These amides have been used to cure com. available,  
unmodified, liq. epoxy resins. The curing behaviors and thermal  
stability of the cured films have been studied. Comparing the  
curing temps. [initial (Ti), peak (Tp), and final (Tf)] and the  
degree of curing, it has been obsd. that using a curing agent contg.  
bulky groups i.e., with a more sterically hindered structure,  
results in higher curing temps. but a lower degree of curing. When  
comparing 2-BU-PHO, which contains a phenoxy group, with 2-BU-PH  
contg. a Ph group, it is interesting to note that the phosphorus  
content in 2-BU-PHO is lower (4.81%) than that of 2-BU-PH (4.93%),  
but the residual wt. percent at 800.degree.C (R800) and also the  
temp. needed to obtain 10% wt. loss (T0.1) is significantly higher  
for 2-BU-PHO cured epoxies. Also the results from 2-BZ-PH, which  
unlike 2-BU-PH contains arom. benzyl groups, indicate that the  
aromaticity of the curing agent does not affect the thermal  
stability of the cured polymer film.

IT **7664-41-7, Ammonia**, reactions  
(starting material; prepn. of phosphoramidate and phosphonamide  
crosslinking agents for fire-resistant epoxy resins)

RN 7664-41-7 HCAPLUS

CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH3

CC 37-6 (Plastics Manufacture and Processing)

Section cross-reference(s): 25, 29

IT Crosslinking agents

**Fireproofing agents**

(prepn. of phosphoramidate and phosphonamide crosslinking agents for fire-resistant epoxy resins)

IT 100-46-9, Benzylamine, reactions 108-95-2, Phenol, reactions

109-73-9, Butylamine, reactions 824-72-6, **Phenylphosphonic dichloride 7664-41-7, Ammonia, reactions**

10025-87-3, Phosphoric trichloride

(starting material; prepn. of phosphoramidate and phosphonamide crosslinking agents for fire-resistant epoxy resins)

L52 ANSWER 3 OF 15 HCAPLUS COPYRIGHT 2004 ACS on STN

2000:775327 Document No. 134:282050 Studies on some **flame retardants** for cotton; Part V: finishing products based on chloromethyl **phosphonic dichloride-diethylamine**

adduct. Abdel-Mohdy, Fikry A.; Nawab, Galal A. M. (Textile Research Division, National Research Centre, Cairo, Egypt). Journal of the Textile Association, 60(3), 121-128 (English) 1999. CODEN: JTXAA9. ISSN: 0368-4636. Publisher: K. P. Publisher.

AB When chloromethyl **phosphonic dichloride** (I) was reacted with diethylamine in 1:2 ratio in presence of triethylamine, the chloromethyl phosphonic acid diamide (II) was obtained. When II was reacted with **NH3**, the aminomethyl phosphonic acid diamide (III) was formed. Hydroxymethylation of III was carried out. Compds. I and II were also made to react with diethanolamine and ethanolamine to give the corresponding hydroxy phosphonamides. The structures of the prepd. compds. were confirmed by phys. tools. The prepd. compds. were applied to cotton fabrics using various methods. The finished fabrics were monitored for N and phosphorus content as well as flammability. Results show all the prepd. amides can be successfully used as **flame retardants** for cotton fabrics.

CC 40-9 (Textiles and Fibers)

ST chloromethyl **phosphonic dichloride** diethylamide **flame retardant** cotton

IT Fabric finishing

(agents; **flame retardant** finishing products

based on chloromethyl **phosphonic dichloride**

-diethylamine adduct (derivs.) for cotton fabrics)

IT Textiles

- (cotton; **flame retardant** finishing products based on chloromethyl **phosphonic dichloride** -diethylamine adduct (derivs.) for cotton fabrics)
- IT **Fireproofing agents**  
(**flame retardant** finishing products based on chloromethyl **phosphonic dichloride** -diethylamine adduct (derivs.) for cotton fabrics)
- IT 110507-54-5, Cellulose-glycidyl methacrylate graft copolymer (fabric precursor; **flame retardant** finishing products based on chloromethyl **phosphonic dichloride**-diethylamine adduct (derivs.) for cotton fabrics)
- IT 25780-08-9P 331412-45-4P 331412-46-5P 331412-47-6P 333362-58-6P  
(**flame retardant** finishing products based on chloromethyl **phosphonic dichloride** -diethylamine adduct (derivs.) for cotton fabrics)
- IT 109-89-7, Diethylamine, reactions 141-43-5, Ethanolamine, reactions 1983-26-2, Chloromethyl **phosphonic dichloride**  
(**flame retardant** finishing products based on chloromethyl **phosphonic dichloride** -diethylamine adduct (derivs.) for cotton fabrics)

L52 ANSWER 4 OF 15 HCAPLUS COPYRIGHT 2004 ACS on STN

X 2000:743424 Document No. 134:253638 Studies on some **flame retardants** for cotton; Part V: Finishing products based on chloromethyl **phosphonic dichloride**-diethylamine.  
Abdel-Mohdy, Fikry A.; Nawar, Galal A. M. (Textile Research Division, National Research Centre, Dokki, Egypt). Book of Papers - International Conference & Exhibition, AATCC 85-94 (English) 1999. CODEN: BPIAEQ. ISSN: 0892-2713. Publisher: American Association of Textile Chemists and Colorists.

- AB Chloromethyl **phosphonic dichloride** (I) reacted with diethylamine in 1:2 ratio in presence of triethylamine, the chloromethyl phosphonic acid diamide (II) was obtained. When II reacted with **ammonia**, the aminomethyl phosphonic acid diamide (III) was formed. Hydroxymethylation of compd. III was carried out. Compds. I and II were also subjected to react with diethanolamine and ethanolamine to give the corresponding hydroxy phosphonamides, resp. The structures of the prepd. compds. were confirmed by phys. tools. The prepd. compds. were applied to cotton fabrics using various methods. The finished fabrics were monitored for nitrogen and phosphorus contents as well as flammability.
- CC 40-9 (Textiles and Fibers)
- ST fabric **fireproofing agent chloromethylphosphonic dichloride** diethylamine; cotton fabric **fireproofing chloromethylphosphonic dichloride** diethylamine

- IT Textiles  
(cotton; **fireproofing** agents based on chloromethyl  
**phosphonic dichloride**-diethylamine for cotton  
fabrics)
- IT **Fireproofing** agents  
Flammability  
(**fireproofing** agents based on chloromethyl  
**phosphonic dichloride**-diethylamine for cotton  
fabrics)
- IT 106-91-2DP, Glycidyl methacrylate, polymers with cotton fabrics,  
graft  
(**fireproofing** agents based on chloromethyl  
**phosphonic dichloride**-diethylamine for cotton  
fabrics)
- IT 50-00-0, Formaldehyde, reactions 109-89-7, Diethylamine, reactions  
111-42-2, Diethanolamine, reactions 141-43-5, Ethanolamine,  
reactions  
(**fireproofing** agents based on chloromethyl  
**phosphonic dichloride**-diethylamine for cotton  
fabrics)
- IT 1983-26-2P, **Chloromethylphosphonic dichloride**  
25780-08-9P 331412-45-4P 331412-46-5P 331412-47-6P  
331412-48-7P  
(**fireproofing** agents; **fireproofing** agents  
based on chloromethyl **phosphonic dichloride**  
-diethylamine for cotton fabrics)

X L52 ANSWER 5 OF 15 HCAPLUS COPYRIGHT 2004 ACS on STN  
1983:216721 Document No. 98:216721 Fire-resistant compositions and  
composites. Byrd, Norman R.; Peek, Daniel C. (McDonnell Douglas  
Corp., USA). U.S. US 4364991 A 19821221, 9 pp. (English). CODEN:  
USXXAM. APPLICATION: US 1980-190741 19800925.

AB Fire-resistant composites or laminates, capable of withstanding  
2000.degree.F flame temp., are prep'd. by incorporating 5-40 parts  
phosphorylated amide (R2R1N)P(X)R (I; X = O, S; R = H, alkyl,  
cycloalkyl, aryl, halogenated alkyl and aryl, heterocyclic; R1,R2 =  
H, alkyl, aryl cycloalkyl, amino, alkylamino, arylamino, aliph.  
acyl, aroyl) into 100 parts resin such as polyimide, epoxy,  
polybenzimidazole, polyester, polyquinoxaline, phenolic, or silicone  
to give a homogeneous compn. The cured compn. is permanently bonded  
to a structural substrate to form a composite structure. When  
subjected to a flame temp. .gtoreq.2000.degree.F, the composite  
forms a resin char of reduced thermal cond. which holds the  
laminating fibers together and maintains the structural stability  
and integrity of the laminate. Thus, a mixt. of 30 g I(X = O; R =  
Ph; R1,R2 = NH2) [4707-88-4], which was prep'd. by treating  
phenylphosphonic dichloride with NH3  
gas, and 135 g polyimide varnish (Skybond 703) was used to

impregnate fiberglass cloth. After curing, the resin-impregnated glass cloth laminate was subjected to a Meeker burner flame at 2000.degree.F for 15 min, exhibiting no burn-off area on the back face and a backside temp. 500.degree.F. The polyimide-glass fabric laminate without I showed a large burn-off area on the back resulting from almost complete volatilization of resin and considerable delamination and a backside temp. 1200.degree.F.

NCL 428265000

CC 38-2 (Plastics Fabrication and Uses)

ST fire resistant resin impregnated fiberglass; phosphorylated amide **fireproofing** polyimide; ablative polyimide glass fiber laminate

L52 ANSWER 6 OF 15 HCAPLUS COPYRIGHT 2004 ACS on STN

1981:533027 Document No. 95:133027 Synthesis of new water-soluble phosphorus- and nitrogen-containing **fireproofing**

compounds. Zubkova, N. S.; Tyuganova, M. A.; Shablygin, M. V.; Kireev, V. V. (Mosk. Tekst. Inst., Moscow, USSR). Izvestiya Vysshikh Uchebnykh Zavedenii, Khimiya i Khimicheskaya Tekhnologiya, 24(4), 471-4 (Russian) 1981. CODEN: IVUKAR. ISSN: 0579-2991. OTHER SOURCES: CASREACT 95:133027.

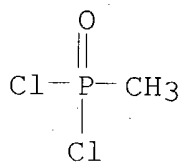
AB Treating  $\text{RP(O)Cl}_2$  with  $\text{MSCN}$  ( $\text{M} = \text{K}, \text{Na}, \text{NH}_4$ ) gave  $\text{RP(O)(NCS)}_2$  which on treatment with  $\text{NH}_3$  gave title compds.  $\text{RP(O)(NH}_2\text{)(NHCSNH}_2\text{)}$  and  $\text{RP(O)(NHCSNH}_2\text{)}_2$  ( $\text{R} = \text{Me}, \text{Et}$ ). These compds. are useful as **fireproofing** agents for cellulose and synthetic fibers.

IT 676-97-1 1066-50-8

(reaction of, with thiocyanate deriv.)

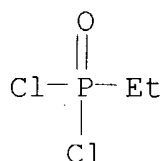
RN 676-97-1 HCAPLUS

CN Phosphonic dichloride, methyl- (8CI, 9CI) (CA INDEX NAME)



RN 1066-50-8 HCAPLUS

CN Phosphonic dichloride, ethyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)



CC 29-7 (Organometallic and Organometalloidal Compounds)  
Section cross-reference(s): 39

ST phosphonodiamidate thiocarbamoyl; **fireproofing** agent  
phosphonodiamidate

IT **Fireproofing** agents  
(thiocarbamoyl phosphonodiamidates)

IT 4519-65-7P 79081-03-1P  
(prepn. and reaction of, with ammonia)

IT 676-97-1 1066-50-8  
(reaction of, with thiocyanate deriv.)

L52 ANSWER 7 OF 15 HCAPLUS COPYRIGHT 2004 ACS on STN

X 1979:55754 Document No. 90:55754 Fire-resistant electric insulators.  
Yoshioka, Akira; Seki, Toshiyuki; Iwasa, Yoshisuke (Sumitomo  
Bakelite Co., Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 53108180  
19780920 Showa, 8 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP  
1977-22251 19770303.

AB Base sheets are impregnated with blends of 65-95% phenolic resins  
and 5-35% polyphosphate esters, and the prepregs contg. 29-65 wt.%  
resins are laminated by heat pressing to give fire-resistant elec.  
insulators. Thus, a mixt. of PhOH 295, o-isopropylphenol 277, 37%  
HCHO 584, and 28% NH<sub>3</sub> 12 parts was refluxed 150 min,  
dehydrated, and dild. with MeOH to give a copolymer (I)  
[68874-95-3] soln. A mixt. of benzoguanamine 300, 37% HCHO 385, and  
BuOH 50 parts at pH 9.5 was heated 135 min at 90.degree.,  
dehydrated, and dild. with Me<sub>2</sub>CO to give a copolymer (II)  
[26160-89-4] soln. Paper (140 g/m<sup>2</sup>) was impregnated with a compn.  
of I 100, II 40, and oligomeric hydroquinone-Ph  
phosphorodichloridate copolymer ester with PhOH [68822-07-1] (mol.  
wt. 697) 45 parts (solids) and dried 5 min at 135.degree. to give a  
prepreg. contg. 49% resin. A pile of 8 of those prepregs and  
35-.mu. Cu foil was pressed 40 min at 173.degree. to give an elec.  
insulator-Cu laminate having soldering resistance (260.degree.) >36  
s, flexural strength 11.2 kg/mm<sup>2</sup>, and fire-resistance rating V0.

IC B32B005-02

CC 36-6 (Plastics Manufacture and Processing)

ST phenolic resin elec insulator; fire resistance phenolic resin;  
polyphosphate ester **fireproofing** agent

IT Polyphosphates  
(**fireproofing** agents, for phenolic resin elec.



insulators)

IT **Fireproofing agents**

(polyphosphates, for phenolic resin elec. insulators)

IT 1319-77-3D, reaction products with **phenylphosphonic dichloride**-resorcinol copolymer and bromophenol  
25766-96-5D, reaction products with cresol and bromophenol  
25949-28-4D, reaction products with cresol and bromophenol  
32762-51-9D, reaction products with **phenylphosphonic dichloride**-resorcinol copolymers and cresol 68796-47-4  
68821-93-2 68822-07-1 68874-96-4

(**fireproofing** agents, for phenolic resin elec. insulators)

L52 ANSWER 8 OF 15 HCAPLUS COPYRIGHT 2004 ACS on STN

X 1978:548108 Document No. 89:148108 Compositions and process for imparting durable flame resistance to cellulosic textiles. Valko, Emery I.; Tesoro, Giuliana C.; Olds, Walter F. (United States Dept. of Agriculture, USA). U.S. US 4092108 19780530, 13 pp. (English). CODEN: USXXAM. APPLICATION: US 1977-758312 19770110.

AB The reaction products of a phosphonic acid deriv. and **NH3**, **H2NNH2**, or amines gives compds. which **fireproof** cellulosic textiles, are durable to laundering, and are effective at about half the concn. of com. **fireproofing** agents. Thus, 1 mol (chloromethyl)**phosphonic dichloride** [1983-26-2] was treated with 4 mol **H2NNH2** to give a mixt. contg. **ClCH2P(O)(NHNH2)2** [67704-63-6]. A soln. of the mixt. was padded on cotton twill, squeezed, dried, cured at 350.degree.F, and laundered at 140.degree.F to give a fabric with 14% add-on giving a char length of 2 min (DOC-FF 3-71) which increased to 5 in after 5 launderings with bleach at 140.degree.F.

IT **7664-41-7**, reactions

(reaction of, with **alkylphosphonic dichloride** derivs.)

RN 7664-41-7 HCAPLUS

CN Ammonia (8CI, 9CI) (CA INDEX NAME)

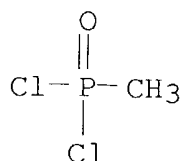
**NH3**

IT **676-97-1 4708-04-7**

(reaction of, with hydrazine)

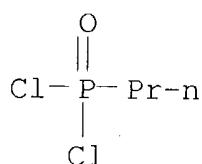
RN 676-97-1 HCAPLUS

CN Phosphonic dichloride, methyl- (8CI, 9CI) (CA INDEX NAME)



RN 4708-04-7 HCAPLUS

CN Phosphonic dichloride, propyl- (6CI, 7CI, 8CI, 9CI) (CA INDEX NAME)



IC D06M013-26

NCL 008116000P

CC 39-10 (Textiles)

Section cross-reference(s): 29

ST **fireproofing** agent phosphonate deriv; cotton  
**fireproofing**; hydrazide phosphonate **fireproofing**  
 agent; amine phosphonate **fireproofing** agent

IT **Fireproofing** agents

(phosphonic acid amine derivs., for cotton textiles)

IT 465-60-1 4759-30-2 6326-70-1 19233-71-7 19280-61-6  
 58980-83-9 67704-59-0 67704-60-3 67704-61-4 67704-62-5  
 (**fireproofing** agents, for cotton)

IT 56818-66-7 67704-54-5 67704-55-6 67704-56-7 67704-57-8  
 67704-58-9 67704-63-6

(fireproofing agents, for cotton, durable to laundering)

IT 7664-41-7, reactions

(reaction of, with alkylphosphonic dichloride derivs.)

IT 676-98-2

(reaction of, with ammonia)

IT 676-97-1 1066-52-0 4708-04-7 5284-09-3  
 17982-64-8

(reaction of, with hydrazine)

IT 74-89-5, reactions

(reaction of, with methylphosphonic dichloride )

X

1978:24188 Document No. 88:24188 A chlorine bleach resistant durable **flame-retardant** finish for cotton based on chloromethylphosphonic diamide. Margavio, Matthew F.; Pepperman, Armand B., Jr.; Constant, Lloyd A.; Gonzales, Elwood J.; Vail, Sidney L. (SRRC, USDA, New Orleans, LA, USA). Journal of Fire Retardant Chemistry, 4(3), 192-206 (English) 1977. CODEN: JFRCDQ. ISSN: 0362-1693.

AB Medium wt. 100% cotton fabrics were made durably **flame retardant** by treatment with a 40% aq. chloromethylphosphonic diamide (I) [6326-70-1] soln. contg. no catalyst or other additives. A conventional pad-dry-cure procedure provided finishes durable to 50 home laundry cycles with or without Cl bleach. The fabric retained a good hand and no HCHO was available for release during the cure. Strength losses were acceptable compared to that of conventional **flame retardant** finishes. The slight yellowing that occurred could be removed by Cl bleaching. The finish was not suitable for lightwt. fabrics when applied by the std. pad-dry-cure procedure. A double process in which the straight pad-dry-cure application of I was followed by a pad-dry-cure topping treatment with 5% trimethylolmelamine [1017-56-7] provided cotton printcloth with a **flame retardance** durable to 50 home launderings.

IT 7664-41-7, reactions  
(reaction of, with **chloromethylphosphonic dichloride**)

RN 7664-41-7 HCAPLUS

CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH3

CC 39-10 (Textiles)

ST **fireproofing** cotton chloromethylphosphonic diamide;  
chlorine bleach resistant **fireproofing**

IT Bleaching agents  
(chlorine, **fireproofing** agents for cotton textiles  
resistant to, chloromethylphosphonic diamide as)

IT **Fireproofing** agents  
(chloromethylphosphonic diamide, chlorine bleach-resistant, for  
cotton textiles)

IT 1017-56-7  
(cotton textile **fireproofing** by chloromethylphosphonic  
diamide and, for improved resistance to chlorine bleaches)

IT 6326-70-1  
(**fireproofing** agents, chlorine bleach-resistant, for  
cotton textiles)

IT 1983-26-2

(reaction of, with ammonia)  
IT 7664-41-7, reactions  
(reaction of, with chloromethylphosphonic  
dichloride)

L52 ANSWER 10 OF 15 HCAPLUS COPYRIGHT 2004 ACS on STN

X 1968:79601 Document No. 68:79601 **Flameproofing** cellulosic  
textiles. (Badische Anilin- & Soda-Fabrik AG). Neth. Appl. NL  
6615460 19670505, 9 pp. (Dutch). CODEN: NAXXAN. PRIORITY: DE  
19651104.

AB Nontoxic and hydrolysis stable **flameproofings** were formed  
on cellulosis textiles by treating them with an aq. soln. contg.  
100-500 g./l. chloromethylphosphonic acid diamide (I) or  
chloromethylphosphonic acid bis(methylamide) (II) in such a manner  
that the textiles contain 20-40% on their dry wt. of the P-compd.  
The fabrics were then dried and heated at 140-70.degree.. I and II  
were prepd. by reacting **chloromethylphosphonic acid**  
**dichloride** (III) with excess **NH3** or **MeNH2** in  
**CH2Cl2**. Thus, a soln. of 84 g. III in 250 g. **CH2Cl2** was added  
dropwise at .apprx.-20.degree. to a soln. of 40 g. **NH3** in  
1000 g. **CH2Cl2**. The mixt. was refluxed for 1 hr., and cooled to  
room temp., to give 116 g. of a cryst. ppt., which was boiled with  
alc., to give 58 g. I, m. 108-9.degree.. Similarly prepd. was II,  
m. 71.degree.. A cotton fabric was impregnated for 10 min. in a  
bath contg. 300 g./l. I, dried at 80.degree., and heated for 15 min.  
at 150-60.degree..

IC C07F

CC 39 (Textiles)

ST PHOSPHORUS CELLULOSE TEXTILES; CELLULOSE TEXTILES **FLAMEPROOF**  
; TEXTILES **FLAMEPROOF** CELLULOSE; **FLAMEPROOF**  
CELLULOSE TEXTILES

IT Textiles

(**fireproofing** cotton, with chloromethylphosphonic acid  
bis(methylamide) and diamide)

IT **Fireproofing**

(of textiles with chloromethylphosphonic acid bis(methylamide)  
and diamide)

IT 6326-70-1P 19318-76-4P

(manuf. of, and cotton textile **flameproofing** with)

X L52 ANSWER 11 OF 15 HCAPLUS COPYRIGHT 2004 ACS on STN

1968:39621 Document No. 68:39621 Phosphinic amides. Nielsen, Morris  
L.; Greenley, Robert Z. (Monsanto Research Corp.). U.S. US 3359276  
19671219, 7 pp. (English). CODEN: USXXAM. APPLICATION: US  
19660901.

GI For diagram(s), see printed CA Issue.

AB The title compds. (I), where Z = imidazol-1-yl or an aromatic  
hydrocarbyl radical and R and R' = a hydrocarbyl radical or H were

prepd. The compds. were used as **flame-retarding** agents for cellulosic fibers and textiles or were treated with 5,5'-bibenzimidazoles to form solid, resinous polymers. Thus, a soln. of 17 g. imidazole in 400 ml. tetrahydrofuran (II) was treated with a soln. of 12.2 g. **phenylphosphonic dichloride** in 75 ml. II, under N and refluxed for 3 hrs. to yield diimidazol-1-ylphenylphosphine oxide (III), m. 98-100.degree.. A mixt. of 0.026 mole III and 0.026 mole PhNHMe was heated, under N, to 235.degree. over a period of 3 hrs. to yield 5.9 g. P-imidazol-1-yl-N-methyl-N,P-diphenylphosphinic amide, m. 132-9.degree.. P-Imidazol-1-yl-N,N,P-triphenylphosphinic amide, m. 178-86.degree., was prepd. by treating III with Ph2NH. A soln. of 17 g. imidazole in 400 ml. II was treated with a soln. of 14 g. methylphenylphosphoramidic dichloride in 75 ml. II to yield P,P-diimidazol-1-yl-N-methyl-N phenylphosphinic amide (IV), m. 122-3.degree.. P,P-Diimidazol-1-yl-N-phenylphosphinic amide (V), m. 163-4.degree., was prepd. by treating triimidazol-1-ylphosphine oxide with PhNH2. V was also prepd. by treating sodium imidazolid with phenylphosphoramidic dichloride. A mixt. of 7 g. 3,3',4,4'-biphenyltetramine tetrachloride, 17.4 g. formic acid, and 38 ml. 5N HCl was refluxed for 0.5 hr. and then poured into 55 ml. concd. **NH4OH** and ice to yield 5,5'-bibenzimidazole (VI), m. 265-7.degree.. A mixt. of 4.306 g. IV and 3.510 g. VI was heated to 255.degree. to yield 5.472 g. VII. The polymer gave a hard, well-dimensioned, molded test specimen when molded at 12,000 psi. and a temp. of 300-50.degree..

NCL 260309000

CC 28 (Heterocyclic Compounds (More Than One Hetero Atom))

ST IMIDAZOLYLPHOSPHINIC AMIDES; PHOSPHINIC AMIDES IMIDAZOLYL;  
**FLAME RETARDANTS** CELLULOSES; PREIGNITION  
INHIBITORS; AMIDES IMIDAZOLYLPHOSPHINIC; CELLULOSES **FLAME**  
**RETARDANTS**; POLYBENZIMIDAZOLES P

L52 ANSWER 12 OF 15 HCAPLUS COPYRIGHT 2004 ACS on STN

1961:90256 Document No. 55:90256 Original Reference No. 55:17031b-d  
Fibrous phosphonomethyl ethers of cellulose. Drake, George L., Jr.;  
Reeves, Wilson A.; Guthrie, John D. (U.S. Dept. of Agriculture). US  
2979374 19610411 (Unavailable). APPLICATION: US .

AB Treatment of cellulosic material with an aq. soln. of an alkali  
metal salt of chloromethylphosphonic acid and an excess of NaOH  
gives a phosphonomethyl ether of cellulose or decreased flammability  
and high cation-exchange capacity. For example, cotton shetting was  
padded by using 2 dips and nips and light squeeze-roll pressure with  
a soln. contg. 60 parts H2O, 25 parts NaOH, and 15 parts  
chloromethyl **phosphonic dichloride** to a wet  
pick-up of 143%. Wet fabric was heated in a forced draft oven for  
30 min. at 110.degree., washed, and dried. The product contained  
1.99% P, had an ion-exchange capacity of 1.179 meq./g., and passed

the 90.degree. angle flame test with a slight afterglow. The NH<sub>4</sub> salt could be formed by soaking the above fiber for 15 min. in 5% HCl, washing acid free, then soaking in 5% NH<sub>4</sub>OH. The fabric then contained 1.95% P, 0.78% N, passed the vertical flame test satisfactorily, and had no afterglow in the 180.degree. angle test.

CC 25 (Dyes and Textiles)

IT **Fireproofing**

(of cotton textiles, by cellulose phosphonomethyl ether formation)

L52 ANSWER 13 OF 15 HCAPLUS COPYRIGHT 2004 ACS on STN

1958:15873 Document No. 52:15873 Original Reference No. 52:2888a-c

~~Dialkyl~~ alkyl chloromethanephosphonates. (Victor Chemical Works). GB 783018 19570918 (Unavailable). APPLICATION: GB .

X AB (RO)2P(O)CH<sub>2</sub>Cl (I), where R is an alkyl group, were prep'd. from Cl<sub>2</sub>P(O)CH<sub>2</sub>Cl (II) with the appropriate alc. in vacuo below 30.degree.C. Thus, to 468.0 g. BuCH<sub>2</sub>CH<sub>2</sub>OH (III) at 20 mm. was added dropwise 100.5 g. II in 1 hr. 11 min., the temp. being kept at 25-30.degree.C. The flask was then pumped at 20 mm. 2 hrs. at 25-30.degree.C., 15 min. each at 35.degree.C., 45.degree.C., and 50.degree.C., then 1.5 hrs. at 4 mm. with continued heating to remove the remaining HCl. The excess III was distd., the crude product washed with 300 ml. 5% NaHCO<sub>3</sub>, then distd., giving 93.5% I (R = BuCH<sub>2</sub>CH<sub>2</sub>) (IV), b.p. 155.degree., n<sub>25D</sub> 1.4509, d. 0.9949. Similarly, 81.4% I (R = Pr) (V), b.p. 72-82.degree., n<sub>25D</sub> 1.4395, d. 1.2167, and the following I were prep'd. (R, b.p./mm., n<sub>25D</sub>, and d. given): Bu (VI), 101.degree./1.5, 1.4418, 1.0782; Am, 122-30.degree./1.0-2.0, 1.4443, 1.0373; Me(CH<sub>2</sub>)<sub>5</sub>, 124-38.degree./2.0, 1.4462, 1.0193; isooctyl (VII), 155.degree./1.0, 1.4509, 0.9891; Me(CH<sub>2</sub>)<sub>8</sub>, 165.degree./1.0, 1.4570, 0.9736; and Me(CH<sub>2</sub>)<sub>9</sub>, 201.degree./1.0, 1.4545, 0.9698. The following I had viscosities (centipoise at 30.degree.C., 105.degree.F., and 212.degree.F.) and surface tensions (dynes/sq. cm. at 30.degree.C.): V, 12, 4.1, 1.6, 26.0; VI, 16, 4.65, 1.8, 27.3; IV, 21, 9.85, 2.7, 28.1; and VII, 19, 11.1, 3.4, 27.5. The I are useful as plasticizers for CH<sub>2</sub>:CHCl-CH<sub>2</sub>:CHOAc copolymers and cellulose acetate, nitrocellulose, and ethyl cellulose.

IT **7664-41-7, Ammonia**

(reactions of, with alkyl halides)

RN 7664-41-7 HCAPLUS

CN Ammonia (8CI, 9CI) (CA INDEX NAME)

NH<sub>3</sub>

CC 10 (Organic Chemistry)

- IT Fire-resistant materials, **Flame-retardant**  
materials  
(dialkyl (chloromethyl)phosphonates as)
- IT Alcohols  
(reactions of, with (chloromethyl)**phosphonic**  
**dichloride**)
- IT Alkyl halides  
(reactions with **NH3**)
- IT 1983-26-2, **Phosphonic dichloride**,  
(chloromethyl)-  
(reaction with alcs.)
- IT **7664-41-7, Ammonia**  
(reactions of, with alkyl halides)

L52 ANSWER 14 OF 15 HCAPLUS COPYRIGHT 2004 ACS on STN

X 1957:9201 Document No. 51:9201 Original Reference No. 51:1878a-i  
Phenoxymethylphosphonic acids and phosphonic acid ion-exchange  
resins. Walsh, Edward N.; Beck, Thomas M.; Toy, A. D. F. (Victor  
Chem. Works, Chicago Heights, IL). Journal of the American Chemical  
Society, 78, 4455-8 (Unavailable) 1956. CODEN: JACSAT. ISSN:  
0002-7863.

AB  $\text{ClCH}_2\text{P}(\text{O})(\text{OH})_2$  (I) in  $\text{H}_2\text{O}$  neutralized with 1 equiv.  $\text{NaOH}$  gave  
 $\text{ClCH}_2\text{P}(\text{O})(\text{OH})\text{ONa}$ , white solid; neutralization of aq. or alc. I with  
2 equivs.  $\text{NaOH}$  yielded 98%  $\text{ClCH}_2\text{P}(\text{O})(\text{ONa})_2$  (II), white hygroscopic  
solid. The appropriate Na phenolate in the phenol, prepd. by  
heating 16.0 g.  $\text{NaOH}$  with 200 g. of the phenol, stirred 5 hrs. at  
160.degree. with 69.8 g. II, the mixt. cooled and extd. with 300 cc.  
 $\text{EtOH}$ , the residual Na salt acidified with 200 cc. concd.  $\text{HCl}$  and  
extd. with  $\text{Et}_2\text{O}$ , and the ext. concd. on the steam bath gave the  
corresponding  $\text{ArOCH}_2\text{P}(\text{O})(\text{OH})_2$  (III) (Ar, g. yield, and m.p. given):  
Ph (IV), 65.4, 141-2.degree.; o-MeC<sub>6</sub>H<sub>4</sub>, 73.5, 157-8.degree.;  
m-MeC<sub>6</sub>H<sub>4</sub> (V), 80.2, 115-17.degree.; p-Me<sub>3</sub>CC<sub>6</sub>H<sub>4</sub>, 68.0, 132-4.degree.;  
p-BrC<sub>6</sub>H<sub>4</sub>, 84.0, 166.8.degree.; p-ClC<sub>6</sub>H<sub>4</sub> (VI), 76.2, 156-7.degree.;  
2,4-Cl<sub>2</sub>CHH<sub>3</sub>, 73.7, 141-2.degree.; 2,4,5-Cl<sub>3</sub>C<sub>6</sub>H<sub>2</sub>, 89.9,  
175-8.degree.. I (52.2 g.) added to the product from 50 g.  $\text{NaOH}$  and  
200 g.  $\text{PhOH}$  in 150 cc. xylene, cooled, dild. with 200 cc.  $\text{H}_2\text{O}$ , and  
neutralized with  $\text{HCl}$ , the aq. layer decanted, washed with  $\text{C}_6\text{H}_6$ ,  
acidified with 200 cc. concd.  $\text{HCl}$ , and extd. with  $\text{Et}_2\text{O}$ , and the ext.  
worked up yielded 66.0 g. IV, m. 140-2.degree. (from  $\text{Et}_2\text{O}$  and  $\text{C}_6\text{H}_6$ ).  
The III are dibasic acids, sol. in  $\text{H}_2\text{O}$ ,  $\text{Et}_2\text{O}$ , and  $\text{EtOH}$ , sparingly  
sol. in  $\text{C}_6\text{H}_6$  and hexane. IV heated with  $\text{PCl}_5$  yielded 20%  
 $\text{PhOCH}_2\text{P}(\text{O})\text{Cl}_2$ , b2 120-30.degree.,  $n_{\text{D}}^{25}$  1.5470. IV (10.0 g.) in 20.0  
cc.  $\text{H}_2\text{O}$  refluxed 20 min. with 6.0 cc. 37% aq.  $\text{CH}_2\text{O}$ , the excess  $\text{H}_2\text{O}$   
removed at 110.degree., and the residual viscous gum treated with  
1.0 cc. aq.  $\text{CH}_2\text{O}$  and baked 16 hrs. at 140.degree. yielded 11.0 g.  
brittle, hard, insol. resin (VII); the gummy condensation product  
baked 3 hrs. at 140.degree. gave a fusible,  $\text{H}_2\text{O}$ -sol. resin;  
extension of the baking period beyond 4 hrs. gave solid, insol.,

infusible resins. VII ground, the 20-60-mesh material washed with 2N NaOH, H<sub>2</sub>O, and HCl, and dried at 110.degree., a 1-g. sample treated with excess 0.1N NaOH, and the unreacted alkali titrated showed that 9.66 milliequivs. Na<sup>+</sup> were exchanged. Di-Na salt (4.64 g.) (VIII) of IV in 20 cc. H<sub>2</sub>O and 5.0 g. 37% aq. CH<sub>2</sub>O refluxed 1 hr. and evapd. on the steam bath yielded 6.2 g. HOCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>OCH<sub>2</sub>P(O)(ONa)<sub>2</sub> (IX), white solid. IX did not resinify at 160.degree.. VIII (4.64 g.) in 20 cc. H<sub>2</sub>O and 5.0 g. 37% aq. CH<sub>2</sub>O refluxed 1 hr., cooled, acidified with 10 cc. concd. HCl, and extd. with Et<sub>2</sub>O, and the ext. worked up gave 4.5 g. viscous, gummy oil contg. 11.8% P; this oil baked overnight at 140.degree. gave a hard, brittle resin with ion-exchange properties; it contained 14.9% P. V (30.3 g.) in 100 cc. H<sub>2</sub>O and 150 cc. 37% aq. CH<sub>2</sub>O, refluxed 2 hrs. and evapd. by slow heating to 115.degree. gave a viscous sirup which baked 16 hrs. at 150.degree. yielded 33.0 g. hard, brittle resin (X); the 20-60-mesh material had an exchange capacity of 8.6 milliequivs./g. PhOH (20.0 g.) and 5.0 g. IV in 210 g. 10.6% aq. CH<sub>2</sub>O refluxed 7 hrs. and the resulting white solid baked 16 hrs. at 140.degree. gave an amber solid, contg. 3.2% P and having ion exchange properties. VI (10.0 g.) in 20 cc. H<sub>2</sub>O and 6.0 cc. 37% aq. CH<sub>2</sub>O refluxed 4 hrs. and concd. by heating 110.degree., and the tacky residue baked 16 hrs. at 140.degree. gave a fusible resin, sol. in H<sub>2</sub>O. IV (18.8 g.) in 50 cc. H<sub>2</sub>O refluxed 2 hrs. with 10.0 g. paraformaldehyde, the resulting clear soln. cooled to 25.degree., neutralized with NH<sub>4</sub>OH, treated with 15.0 g. trimethylolmelamine (Resloom HP) in 50 cc. H<sub>2</sub>O, refluxed 1 hr., and allowed to stand deposited a solid resin contg. phosphonic acid groups; the soln. padded on cloth and the impregnated cloth heated 10 min. at 140.degree. imparted flame resistance; the cloth treated with Na salts loses its flame resistance which is restored by treatment with NH<sub>4</sub>OH; the flame resistance can also be restored with dil. acids. The titration curves of VII and X are recorded; VII and X behave as dibasic acids having equivalence points at pH 5.0 and 10.2. A column of 24.2 g. VII (232 milliequivs. exchange capacity) was tested for ion exchange properties by passing solns. of know concns. through the column until both the breakthrough point (1% leakage) and the satn. point of the VII had been reached (ionic form of VII, influent, break-through point in % exchange, regenerant, % regeneration given): H, 0.1N NaOH (about 100% vol. increase occurred), 91, 0.1N HCl, 95; Na, 0.005N Ca(NO<sub>3</sub>)<sub>2</sub>, 85, 0.1N HCl, 90 (2N NaCl, 5%); H, 0.50N Ca(NO<sub>3</sub>)<sub>2</sub>, 27, 0.1N HCl, 95; Na, 10N CuSO<sub>4</sub>, 41, 2N NaCl, 1; NH<sub>4</sub>, 0.10N Cu(NH<sub>3</sub>)<sub>4</sub>SO<sub>4</sub>, 106, N NH<sub>4</sub>Cl, 41; Na, 0.10N MgSO<sub>4</sub>, 40, 0.1N HCl, 38 (2N NaCl, 5%).

CC

10 (Organic Chemistry)

IT

Fire-resistant materials, **Flame-retardant**  
materials

(phenoxymethylphosphonic acid condensation products with



tris(hydroxymethyl)melamine)

- IT 28482-42-0, Phosphonic acid, (chloromethyl)-, sodium salt  
 58765-08-5, Phosphonic acid, [(p-tert-butylphenoxy)methyl]-  
 98280-21-8, Phosphonic acid, [(p-bromophenoxy)methyl]- 98551-01-0,  
**Phosphonic dichloride**, phenoxymethyl-  
 101084-89-3, Phosphonic acid, [(2,4,5-trichlorophenoxy)methyl]-  
 101252-88-4, Phosphonic acid, [(2,4-dichlorophenoxy)methyl]-  
 106421-12-9, Phosphonic acid, (o-tolyloxymethyl)- 108624-78-8,  
 Phosphonic acid, [(alpha.-hydroxytolylloxy)methyl]-, disodium salt  
 (prepn. of)

L52 ANSWER 15 OF 15 HCAPLUS COPYRIGHT 2004 ACS on STN

X 1945:28528 Document No. 39:28528 Original Reference No. 39:4619c-g  
 Organic phosphonic acid dichlorides. Hamilton, Lyle A. (E. I. du  
 Pont de Nemours & Co.). US 2365466 19441219 (Unavailable).  
 APPLICATION: US .

AB An aliphatic compd. having an OH and a -PO3H2 group attached to the  
 same C which in turn is attached to a second C carrying a H atom is  
 heated at 125-250.degree.. H2O is split off, forming an  
 .alpha.,.beta.-ethylenically unsatd. hydrocarbon phosphonic acid.  
 To the product is added portionwise PCl5, using 2-2.5 mols. of PCl5  
 per mol. of phosphonic acid. In the initial stages of adding PCl5  
 cooling is advisable. Lower dichlorides, i.e., those having up to 7  
 C atoms are purified by fractional distn. at reduced pressure; the  
 higher ones, i.e., those with 8 C atoms or more, are purified simply  
 by heating in an open vessel. The dichlorides can be converted to  
 highly pure corresponding phosphonic acids by hydrolysis; they can  
 be converted to diesters by causing them to react with an alc. or  
 phenol; or they can be converted to diamides by a reaction with  
 NH3 or an amine. The prepn. of the following is described:  
1-Propene-2-phosphonic dichloride, b32  
 82.5-86.degree.; di-Me 1-propene-2-phosphonate, b1-2 44-6.degree.,  
 n20 1.4340; di-Bu 1-propene-2-phosphonate, b0.25 86-7.degree., n20  
 1.4376; di-Ph 1-propene-2-phosphonate; N,N,N',N'-tetramethyl-1-  
 propene-2-phosphondiamide, b2-3 76-80.degree., n20 1.4735. Other  
 phosphonic acids suitable for these reactions are listed. The  
 dichlorides are valuable as intermediates; the diamides are useful  
 as plasticizers, softening agents, and as addn. agents to  
 lubricating oils; and the diesters are useful as plasticizers,  
**flame retardants**, and petroleum adjuvants.

CC 10 (Organic Chemistry)

IT **Fireproofing**  
 (phosphonic acid esters for)

=> d his full

(FILE 'HOME' ENTERED AT 11:28:43 ON 04 FEB 2004)

L1 FILE 'LREGISTRY' ENTERED AT 11:28:50 ON 04 FEB 2004  
STRUCTURE

L2 FILE 'REGISTRY' ENTERED AT 11:35:29 ON 04 FEB 2004  
L3 50 SEA SSS SAM L1  
STRUCTURE  
D QUE STAT L3

L4 10 SEA SSS SAM L3  
D SCAN

L5 FILE 'LREGISTRY' ENTERED AT 11:40:28 ON 04 FEB 2004  
STRUCTURE

L6 0 SEA SSS SAM L5  
D QUE STAT L3

L7 0 SEA SSS SAM L3

L8 FILE 'REGISTRY' ENTERED AT 11:45:16 ON 04 FEB 2004  
4 SEA SSS SAM L5  
D SCAN  
D QUE STAT L3

L9 10 SEA SSS SAM L3  
D QUE STAT L5

L10 4 SEA SSS SAM L5

FILE 'HCAPLUS' ENTERED AT 11:53:16 ON 04 FEB 2004  
E ZUBKOVA/AU  
E ZUBKOVA N?/AU

L11 52 SEA "ZUBKOVA N S"/AU OR "ZUBKOVA NINA S"/AU OR "ZUBKOVA  
NINA SERGEEVNA"/AU  
E BUTYLKINA N?/AU

L12 22 SEA "BUTYLKINA N G"/AU OR "BUTYLKINA NATALIYA G"/AU OR  
"BUTYLKINA NATALIYA GRIGORIEVNA"/AU

L13 16 SEA L11 AND L12  
E KHALTURINSKY N?/AU  
E KHALTURINSK? N?/AU

L14 137 SEA ("KHALTURINSKI NIKOLAI ALEXANDROVICH"/AU OR "KHALTURI  
NSKII N"/AU OR "KHALTURINSKII N A"/AU OR "KHALTURINSKIJ  
N"/AU OR "KHALTURINSKIJ N A"/AU OR "KHALTURINSKIJ  
NIKOLAI A"/AU OR "KHALTURINSKIJ NIKOLAY A"/AU OR  
"KHALTURINSKY NIKOLAI ALEXANDROVICH"/AU OR "KHALTURISKII  
N A"/AU)

L15 6 SEA L13 AND L14  
D TI 1-6  
E BERLIN A?/AU  
E BERLIN A/AU

L16 1643 SEA "BERLIN A A"/AU OR "BERLIN AL AL"/AU OR ("BERLIN  
ALEKSANDR A"/AU OR "BERLIN ALEXANDER"/AU OR "BERLIN

ALEXANDER AL"/AU OR "BERLIN ALEXANDR ALEXANDROVICH"/AU  
OR "BERLIN ALEXANDRE"/AU OR "BERLIN ALEXANDRE A"/AU)

L17

3 SEA L15 AND L16

FILE 'LCA' ENTERED AT 12:00:51 ON 04 FEB 2004

FILE 'HCAPLUS' ENTERED AT 12:04:25 ON 04 FEB 2004  
SELECT L17 RN 1-3

FILE 'REGISTRY' ENTERED AT 12:04:40 ON 04 FEB 2004

L18

20 SEA (12125-02-9/BI OR 13598-36-2/BI OR 14500-78-8/BI OR  
25038-54-4/BI OR 29037-34-1/BI OR 322764-30-7/BI OR  
48076-62-6/BI OR 57-13-6/BI OR 67-56-1/BI OR 676-97-1/BI  
OR 7727-21-1/BI OR 80-62-6/BI OR 868-77-9/BI OR 9002-88-4  
/BI OR 9003-07-0/BI OR 9003-08-1/BI OR 9003-31-0/BI OR  
9003-53-6/BI OR 9003-55-8/BI OR 919-30-2/BI)  
D SCAN L18

FILE 'LREGISTRY' ENTERED AT 12:05:44 ON 04 FEB 2004  
DISPLAY FIELDFILE 'REGISTRY' ENTERED AT 12:15:31 ON 04 FEB 2004  
D L18 1-20 FIDE  
D L18 RN CN STR 1-20

FILE 'LREGISTRY' ENTERED AT 13:07:14 ON 04 FEB 2004

FILE 'STNGUIDE' ENTERED AT 13:08:05 ON 04 FEB 2004

FILE 'REGISTRY' ENTERED AT 13:08:53 ON 04 FEB 2004  
D QUE STAT L5

L19

4 SEA SSS SAM L5

D SCAN

L20

118 SEA SSS FUL L5

SAVE L20 ANT719/A

FILE 'LREGISTRY' ENTERED AT 13:12:38 ON 04 FEB 2004

FILE 'HCAPLUS' ENTERED AT 13:15:09 ON 04 FEB 2004

L21

43 SEA L20

D TI 1-43

FILE 'REGISTRY' ENTERED AT 13:39:35 ON 04 FEB 2004

E PHOSPHONAMIDIC ACID/CN

E PHOSPHONAMIDIC ACID, METHYL/CN

L22

1 SEA "PHOSPHONAMIDIC ACID, METHYL ESTER"/CN

D STR RN

L23

7 SEA METHYL(W) PHOSPHONAMIDIC(W) ACID

D SCAN  
E PHOSPHONAMIDIC ACID/CN  
E PHOSPHORIC DICHLORIDE/CN  
E AMMONIA/CN  
L24 1 SEA AMMONIA/CN  
D RN  
E AMMONIUM HYDROXIDE/CN  
L25 1 SEA "AMMONIUM HYDROXIDE"/CN  
D RN  
E ETHYLPHOSPHONYL/CN  
L26 1 SEA "ETHYLPHOSPHONYL DICHLORIDE"/CN  
D RN STR  
E PROPYLPHOSPHONYL/CN  
L27 1 SEA "PROPYLPHOSPHONYL DICHLORIDE"/CN  
D RN STR  
E ISOPROPYLPHOSPHONYL/CN  
L28 1 SEA "ISOPROPYLPHOSPHONYL DICHLORIDE"/CN  
D RN STR  
E METHYLPHOSPHONAMIDIC/CN  
L29 1 SEA "METHYLPHOSPHONIC AMIDE"/CN  
D STR RN  
E ETHYLPHOSPHONIC AMIDE/CN  
E ETHYLPHOSPHONIC/CN  
E ETHYLPHOSPHONAMIDIC ACID/CN  
E PHOSPHONIC AMIDE/CN  
E PHOSPHONIC ACID/CN  
L30 3498 SEA (PHOSPHONAMIDIC)  
L31 161 SEA L30 (2A) ETHYL  
L32 145 SEA L31(2A)ACID  
L33 2 SEA (PHOSPHONAMIDIC ACID) (W)ETHYL  
D SCAN  
L34 0 SEA ETHYLPHOSPHONAMIDIC(W)ACID  
L35 1 SEA ETHYLPHOSPHONAMIDIC  
D SCAN

FILE 'LREGISTRY' ENTERED AT 14:02:48 ON 04 FEB 2004  
D COST

FILE 'REGISTRY' ENTERED AT 14:05:01 ON 04 FEB 2004  
E AMIDOPHOSPHONIC ACID/CN

L36 0 SEA "AMIDOPHOSPHONIC ACID"/CN AND ETHYL  
L37 1608 SEA (PHOSPHONAMIDIC(W)ACID) AND ETHYL  
L38 2 SEA (PHOSPHONAMIDIC(W)ACID) (W) ETHYL  
D SCAN  
L39 67 SEA (PHOSPHONAMIDIC(W)ACID) (W)P(W) ETHYL

FILE 'HCAPLUS' ENTERED AT 14:09:01 ON 04 FEB 2004

L40 393 SEA (14500-78-8/RN OR 52030-74-7/RN OR ?PHOSPHONAMIDIC?)

L41 2628 SEA (676-97-1/RN OR 1066-50-8/RN OR 4708-04-7/RN OR  
 1498-46-0/RN OR (?PHOSPHONIC?(2A)(?CHLORIDE)))  
 L42 411808 SEA 7664-41-7/RN OR 1336-21-6/RN OR NH3 OR NH4OH OR  
 AMMONIA# OR (AMMONIUM(W)HYDROXIDE)  
 L43 21 SEA L40 AND L42  
 L44 78 SEA L41 AND L42  
 L45 94 SEA L43 OR L44  
 L46 38995 SEA (FIRE? OR FLAME OR COMBUST? OR DECOMPOSIT? OR  
 PYROLY?) (2A) (INHIBIT? OR HINDER? OR IMPED? OR PROTECT?  
 OR RETARD? OR PROHIBIT? OR PREVENT? OR BLOCK? OR  
 ELIMINAT? OR PROOF)  
 L47 39506 SEA FIREPROOF? OR FLAMEPROOF? OR FIRERETARDANT? OR  
 FLAMERETARDANT? OR FIREPROTECT? OR FLAMEPROTECT?  
 L48 59503 SEA L46 OR L47  
 L49 2964 SEA L40 OR L41  
 L50 214 SEA L49 AND L48  
 L51 15 SEA L45 AND L48  
 D TI 1-15  
 L52 15 SEA L51 NOT L21  
 L53 15 SEA L50 AND L42  
 L54 0 SEA L53 NOT (L21 OR L52)  
 L55 191 SEA L50 AND ((1907-1999)/PY OR (1907-1999)/PRY)  
 L56 151 SEA L55 AND PATENT/DT  
 L57 153079 SEA (7664-41-7/RN OR 1336-21-6/RN OR NH3/TI OR NH4OH/TI  
 OR AMMONIA#/TI OR (AMMONIUM/TI(W)HYDROXIDE/TI))  
 L58 5 SEA L50 AND L57  
 D SCAN  
 L59 0 SEA L58 NOT (L21 OR L52)  
 L60 137690 SEA (7664-41-7/RN OR 1336-21-6/RN OR NH3/ABS OR NH4OH/ABS  
 OR AMMONIA#/ABS OR (AMMONIUM/ABS(W)HYDROXIDE/ABS))  
 L61 399025 SEA (7664-41-7/RN OR 1336-21-6/RN OR NH3/AB OR NH4OH/AB  
 OR AMMONIA#/AB OR (AMMONIUM/AB(W)HYDROXIDE/AB))  
 L62 14 SEA L55 AND L61  
 L63 0 SEA L62 NOT (L21 OR L52)  
 D QUE STAT L5  
 D QUE STAT L5  
 D L15 1-6 CBIB ABS HITSTR HITIND  
 D L21 1 13 14 15 26 CBIB ABS HITSTR HITIND  
 D L52 1-15 CBIB ABS HITSTR HITIND

FILE HOME

FILE LREGISTRY

LREGISTRY IS A STATIC LEARNING FILE

FILE REGISTRY

Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 3 FEB 2004 HIGHEST RN 646026-80-4  
DICTIONARY FILE UPDATES: 3 FEB 2004 HIGHEST RN 646026-80-4

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Experimental and calculated property data are now available. For more information enter HELP PROP at an arrow prompt in the file or refer to the file summary sheet on the web at:

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FILE COVERS 1907 - 4 Feb 2004 VOL 140 ISS 6

FILE LAST UPDATED: 3 Feb 2004 (20040203/ED)

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#### FILE LCA

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This file contains CAS Registry Numbers for easy and accurate substance identification.

#### FILE STNGUIDE

FILE CONTAINS CURRENT INFORMATION.

Anthony 09/786,719

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LAST RELOADED: Jan 30, 2004 (20040130/UP).